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FOR

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FOR

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A NEW APPROACH TO UNDERSTANDING THE SEMICONDUCTOR SURFACE AND **INTERFACES** 

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Report 1 November 1987 through 31 October 1988

a. Papers submitted to refereed Journals (and not yet published):

Superconductivity up to 90 K in a cubic thallium-doped barium cuprate, Z. Iqbal, H. Eckhardt, B. L. Ramakrishna, J. C. Barry, D. C. Vier, S. Schultz, S. B. Oseroff, E. W. Ong, A. Bose, and F. Reidinger, submitted for publication to <u>NATURE</u>, August 1988. (Preprint attached.)

b. Papers published in refereed journals:

Magnetization of the 120-K Tl-Ca-Ba-Cu-O superconductor, A. M. Hermann, Z. Z. Sheng, D. C. Vier, S. Schultz, and S. B. Oseroff, Phys. Rev. B, **37**, #16, 9742 (1988). (Reprint attached.)

c. Books (and sections thereof) submitted for publication:

None

d. Books (and sections thereof) published:

None

e. Patents filed:

None

f. Patents granted:

None

g. Invited presentations at topical or scientific/technical society conferences:

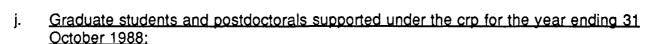
None

h. Contributed presentations at topical or scientific/technical society conferences:

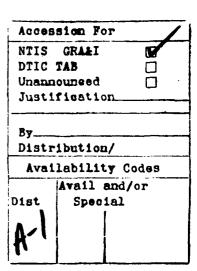
None

i. Honors, Awards/Prizes:

None



Joseph Anderberg, graduate student Dr. David C. Vier, postdoc



#### ANNUAL LETTER REPORT

NOOO14-87-K-0338

For period 1 November 1987 to 31 October 1988

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University of California, San Diego

We have extended our application of electron spin resonance for the study of the silicon metal interface to include Au, Al, and Ag overlayers. In Table I (below) we present representative data for the observed broadening of the ESR linewidth for each of these metals applied to the silicon surface. As can be seen from these data, whereas strong spin coupling was found for Au and Al (comparable to our earlier results for Cu), we found no evidence of coupling for Ag. We believe these differences illustrate the specificity of the Sirmetal atom bond at the interface, whose quantitative measure is one of the goals of this research. We are now in the process of completing the dedicated UHV in-situ ESR system that will enable us to make the desired measurements of the Si-metal coupling at the submonolayer coverage level.

We have continued to search for ESR signals in the new high  $T_c^{\Pi}$  superconductors, and also to apply the low field microwave absorption technique for the purposes of discovering new high  $T_c^{\Pi}$  materials. In particular we have improved the sensitivity of our technique such that we are readily able to detect superconductivity in single crystals as small as  $10^{-6}$  gm. Preprints of articles reporting this effort are enclosed.

TABLE 1

Broadening of ESR Linewidth for Given Metal

<u>METAL</u>	<u>ΔH (Gauss)</u>
Ag	0.0 ± 1
Al	6.2 ± 1
Au	13.4 ± 1.5
Cu	11.0 ± 1.5

#### **Enclosures:**

- (1) A. M. Hermann, Z. Z. Sheng, D. C. Vier, S. Schultz, and S. B. Oseroff, "Magnetization of the 120-K Tl-Ca-Ba-Cu -O superconductor", Phys. Rev. B, **37**, #16, 9742 (1988).
- (2) Z. Iqbal, H. Eckhardt, B. L. Ramakrishna, J. C. Barry, D. C. Vier, S. Schultz, S. B. Oseroff, E. W. Ong, A. Bose, and F. Reidinger, "Superconductivity up to 90K in a cubic thallium-doped barium cuprate", submitted for publication to <u>Nature</u>, August 1988.

Scientific Officer, N00014 Administrative Contracting Officer, N66018 (UCSD)



#### Magnetization of the 120-K Tl-Ca-Ba-Cu-O superconductor

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dc magnetization measurements on the new Tl-Ca-Ba-Cu-O superconductor show superconductivity with an onset temperature as high as 118 K, 24 K higher than that of a high-quality Eu-Ba<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub> sample. The resistive onsets of these new superconductors are near 140 K. The magnetic data show complete diamagnetic flux exclusion and 10-15% Meissner expulsion at 10 K.

Discoveries of 30-K superconductivity in the La-Ba-Cu-O system<sup>1</sup> and 90-K superconductivity in the Y-Ba-Cu-O system<sup>2</sup> have stimulated a worldwide race for higher-temperature superconductors. In spite of many efforts in the past year, stable and reproducible superconductivity has remained at the level of 90 K in the R-Ba-Cu-O system (R equals rare earth). Breakthroughs were recently made in rare-earth-free superconductors by the discoveries of the 90-K Tl-Ba-Cu-O system, 3,4 110-K Bi-Ca-Sr-Cu-O system, 5,6 and 120-K Tl-Ca-Ba-Cu-O system. 7-9 The Tl-Ca-Ba-Cu-O superconducting samples have onset and zero-resistance temperatures much higher than those of the Bi-Ca-Sr-Cu-O system. 7-9 Two superconducting phases have been identified. One has a composition of  $Tl_2Ca_2Ba_2Cu_3O_{10+x}$  (2:2:2:3 phase), and another of Tl<sub>2</sub>CaBa<sub>2</sub>Cu<sub>2</sub>O<sub>8+x</sub> (2:1:2:2 phase, which is similar to Bi<sub>2</sub>CaSr<sub>2</sub>Cu<sub>2</sub>O<sub>8+x</sub>). In this Brief Report we report magnetization and resistance data on the Tl-Ca-Ba-Cu-O samples. The onset temperature of magnetization is 117-118 K, 23 K higher than that of a high-quality EuBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub> sample.

Two samples with nominal compositions Tl<sub>2</sub>Ca<sub>2</sub>Ba-Cu<sub>3</sub>O<sub>9+x</sub> and Tl<sub>2</sub>Ca<sub>4</sub>BaCu<sub>3</sub>O<sub>11+x</sub> used in the present experiments were prepared using Tl<sub>2</sub>O<sub>3</sub>, CaO, and BaCu<sub>3</sub>O<sub>4</sub> in the same batch. 8 Both were heated at 900 °C for 3 min and furnace cooled. The resistance-temperature dependences of these two samples are shown in Fig. 1. The resistance was measured by the standard four-probe technique with silver-paste contacts. These two samples both have onset temperatures near 140 K (defined by the smallest curvature of the resistance-temperature curve 10), midpoint of 127 K, and zero-resistance temperature at 121 K. Zero-resistance data correspond to resistivities less than  $10^{-6} \Omega$  cm. The difference of calcium contents in these two samples does not seem to have significantly changed their superconducting behavior, which depends strongly on preparation conditions of the samples. Note that the Tl<sub>2</sub>Ca<sub>2</sub>BaCu<sub>3</sub>O<sub>9+x</sub> sample consists of approximately 80% of the new 2:2:2:3 superconducting phase.9

Magnetization measurements were performed utilizing a superconducting quantum interference device (SQUID)

magnetometer manufactured by BTI Corp., San Diego, CA. Figures 2 and 3 show dc magnetization (field cooled and zero field cooled) as a function of temperature for an applied field of 1 mT for samples of Tl<sub>2</sub>Ca<sub>2</sub>BaCu<sub>3</sub>O<sub>9+x</sub> and Tl<sub>2</sub>Ca<sub>4</sub>BaCu<sub>3</sub>O<sub>11+x</sub>, respectively, whose resistive behavior is shown in Fig. 1. The Meissner flux expulsion is 10-15% of the diamagnetic flux exclusion; the magnitude of the diamagnetic shielding is large and consistent with complete flux exclusion from the samples at 10 K. This behavior is similar to that observed in Tl-Ba-Cu-O, 4 La-Ba(Sr)-Cu-O, 11 and Y-Ba-Cu-O samples. 12 Explanations suggested to date include existence of a superconducting glass state, 11 anisotropy effects, 13 and superconductivity confined to thick shells around normal grains. 12 At this stage, we hesitate to speculate on differentiation between these causes, and only note that our samples were

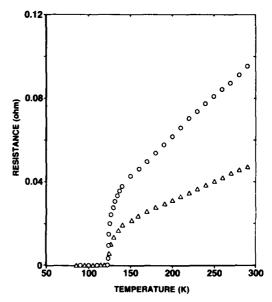


FIG. 1. Resistance-temperature dependences of samples Tl<sub>2</sub>Ca<sub>2</sub>BaCu<sub>3</sub>O<sub>9+x</sub> (triangles) and Tl<sub>2</sub>Ca<sub>4</sub>BaCu<sub>3</sub>O<sub>11+x</sub> (circles).

<u>37</u>

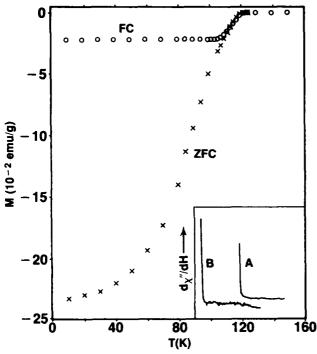


FIG. 2. Field-cooled (FC) and zero-field-cooled (ZFC) magnetization as a function of temperature for a dc field of 1 mT for a sample of  $Tl_2Ca_2BaCu_3O_{9+x}$ . The two data traces in the inset illustrate the sharp onset of superconductivity observed by the microwave technique as described in the text. Sample A is  $Tl_2Ca_2BaCu_3O_{9+x}$  with onset temperature of 117.2 K, and for comparison, data for a sample of  $EuBa_2Cu_3O_{7-x}$  (sample B) with onset temperature of 94.4 K is plotted. Note that the difference in onset is 22.8 K.



In the insets of Figs. 2 and 3, we present data traces for these two samples, and also for a well-prepared EuBa<sub>2</sub>- $Cu_3O_{7-x}$  sample, where the vertical axis represents the dx''/dH signal of an EPR spectrometer. The full details of how we apply this technique will be presented elsewhere, but in brief, we have examined diverse high-T<sub>c</sub> samples including ceramic, powder, single-crystal, and epitaxial films, and find that for properly optimized conditions the temperature dependence of the spectrometer output may be taken as a sensitive indication for the onset of superconductivity. As is seen from the inset of Fig. 2, the onset temperature for the sample A  $(Tl_2Ca_2BaCu_3O_{9+x})$ is 117.2 K, and, for comparison, for sample B (EuBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub>), the corresponding value is 94.4 K (the difference in onset temperature between the Tl-Ca-Ba-Cu-O sample and the Eu-Ba-Cu-O sample is 22.8 K).

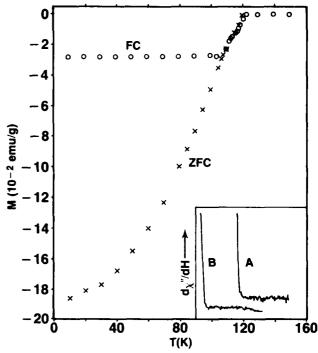


FIG. 3. Field-cooled (FC) and zero-field-cooled (ZFC) magnetization as a function of temperature for a dc field of 1 mT for a sample of  $Tl_2Ca_4BaCu_3O_{11+x}$ . The two data traces in the inset illustrate the sharp onset of superconductivity observed by the microwave technique as described in the text. Sample A is  $Tl_2Ca_4BaCu_3O_{11+x}$  with onset temperature of 118.3 K, and for comparison, data for a sample of  $EuBa_2Cu_3O_{7-x}$  (sample B) with onset temperature of 94.4 K is plotted. Note that the difference in onset temperatures is 23.9 K.

Similarly, from the inset of Fig. 3, the corresponding value for the sample Tl<sub>2</sub>Ca<sub>4</sub>BaCu<sub>3</sub>O<sub>11+x</sub> is 118.3 K, 23.9 K higher than that of the Eu-based sample. These onset temperatures are consistent with those measured by resistance-temperature variations.

In conclusion, the Tl-Ca-Ba-Cu-O samples show a complete diamagnetic exclusion, and 10-15% Meissner expulsion at 10 K. The onset temperature camagnetization drop for the Tl-Ca-Ba-Cu-O samples is 118 K, which is 23.5 K higher than that for a high-quality EuBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub> sample. This onset temperature is consistent with that observed from resistance-temperature variations.

The support of the National Science Foundation (Grant No. NSF-DMR-86-13858) and the U.S. Office of Naval Research (Grant No. ONR-N000014-87-K-0338) is gratefully acknowledged.

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Superconductivity up to 90K in a cubic thallium-doped barium cuprate

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The layered tetragonal Bi-Sr-Ca-Cu $^{1-3}$  and Ti-Ba-Ca-Cu $^{4\cdot5}$  oxides with bulk transition temperatures in the 80-125K range have been the centre of the recent excitement in the field of high  $J_{c}$  superconductivity. Another important development has been the construction of a cubic, non-copper oxide superconductor of composition  $B_{1-x}K_{k}Bi0_{5}^{3}$ , with a  $T_{c}$  onset of 30K. Here we report the results of experiments in the Ti-Ba-Cu oxide precursor system with and without Ca, from which we were able to isolate a cubic phase with a superconducting transition onset which varies with thallium concentration from 30 to 90K. The cubic phase is a thallium-doped barium cuprate of typical composition  $Ba_{0.9-x}TI_{x}^{\times}CuO_{y}$  where x-0.2 as determined by the refinement of single crystal X-ray diffraction data. Superconductivity in this compound apparently occurs, as in  $Ba_{1-x}K_{x}BiO_{3}$ , within the framework of a 3-dimensionally linked array.

In the course of our investigation of the preparation conditions of the layered thallium-calcium-barium-copper oxide superconductors<sup>4,5</sup>, we observed the formation of a metastable, superconducting cubic thallium doped barium cuprate phase under the particular synthesis conditions described here. The cubic phase was observed as the majority fraction (> 85% by volume via X-ray diffraction) in samples prepared from the starting nominal compositions of the metal atoms II:Ba:Cu = 1:2:3 and 1:2:4. The cubic phase is also formed as a 50% volume fraction from a precursor containing Ca in the ratio II:Ba:Ca:Cu = 2:2:2:3.

The best synthesis conditions consisted of firing BaCO $_3$  and CuO in the 2:3 or 2:4 atomic proportions for 25 hrs. near 930°C (or close to the melting point of the starting composition) in air with one intermediate grinding. The pre-formed barium copper oxide was then mixed carefully with the appropriate amount of  $\Pi_2^{0}$ 3 and pressed into pellets at -5kbar pressure. The pellets were placed in an alumina boat lined with gold foil, which was placed in the cold part of a tube furnace under flowing  $\theta_2$ . The reaction zone of the furnace was heated to between 860°C and 865°C, the boat was then pushed into the reaction zone and the temperature stabilized within 3 minutes. The heat treatment was carried out typically for 5 min. followed by removal of

both T1,03 and T1,003 decomposes and liquid phase formation can occur, indicated by asterisks. Because of the better match of the ionic size this was followed by annealing under flowing oxygen at 450°C for 1 hr. down within 5 minutes to at least 400°C under flowing 0,. The powder thus allowing rapid chemical doping of the barium cuprate phase. In the boat to the cold zone of the furnace where it is allowed to cool K-ray diffraction pattern of a sample prepared from a 1:2:3 initial cuprate phase from a 2:2:3 precursor under flowing Ar with thallous carbonate or nitrate instead of  $\Pi_2 \theta_3$  in the precursor composition. starting stoichiometries and temperatures suggest a need for excess thallium in the starting precursor and processing above 800°C where addition, control of the oxygen content of the barium cuprate phase of II with Ba2+, we prepared a few samples of the Il-doped barium precursor appears necessary via flux formation and/or an annealing associated with the minority thallium-rich phase in the sample are volume fraction of the minority phase indicated by asterisks is initial composition containing  ${\rm Tl_2CO_4}$  is shown in Fig. 1(b). The somewhat smaller than in the sample prepared from a 1:2:3 nominal composition containing  $\Pi_2^{0}$ 3. Our experiments with a variety of The X-ray diffraction pattern of a sample prepared from a 2:2:3 composition is shown in Fig. 1(a). The same X-ray pattern was obtained for a sample prepared from a 1:2:4 precursor. Peaks step under Ar.

Electron microprobe analyses in the focussed mode (1 µm) in a scanning electron microscope were carried out for typical preparations from precursor compositions mentioned above. The microprobe analysis of the doped phase showed a Cu:8a atomic ratio that was consistently near 1.4. The Cu:11 atomic ratio in the doped phase was found to range from -5.5 to 7.0.

The samples described above were examined extensively in the high resolution transmission electron microscope at Arizona State University. The micro-crystals were consistently found to be cubic in the imaging and diffraction modes. Typical (100) and (111) images and the corresponding diffraction patterns are shown in Fig. 2. We have also recorded the structure image of the undoped phase and found the intensity distribution of the dark spots (which represent the atomic

positions) and the electron diffraction pattern to be different from that of the thallium-doped structure. The minority component in the samples investigated consisted of a fine grained material which did not correspond to any of the known superconducting layered thallium-barium-copper oxide phases, <sup>4,5</sup> both from the images (which were difficult to obtain because of disorder in this phase) and the electron diffraction patterns. Electron microprobe analyses of the minority phase showed that it is thallium-rich, but there is no unique thallium-barium-copper composition.

prepared from a T1:Ba:Ca:Cu = 2:2:2:3 composition. These crystals and lattice constant of 18.277 A and belongs to the space group Im3m. The are listed in Table 1. A model of the structure which shows hexagonal Single crystal X-ray diffraction analyses were carried out on a involving the Cu (4) and O(6) locations. Hence the oxygen and copper X-ray diffraction data. The electron microprobe data, however, fixes determination is:  $Ba_{0.9-x} Tl_x Cu0_y$ , with x = 0.2. There is disorder those obtained from a CaO-free precursor contained no Ca as shown by In the doped structure, Ba atoms are missing from the fractional coordinates, thermal factors and occupancies at R = 0.05 occupancies and sitting astride the body diagonals, is shown in Fig. 0,0,0 and 1/2,1/2,1/2 positions, and there is substitutional doping 3. The Cu atoms in this position are four-coordinated. This local  $\mathsf{Cu}_6\mathsf{O}_{12}$  rings involving the  $\mathsf{Cu}(1)$  and  $\mathsf{O}(2$  and 3) sites capped by  $\mathsf{Tl}$ structure may provide the key to the observed superconductivity in concentrations are not uniquely determined from the single crystal by thallium at three Ba positions. The structure is cubic with a electron microprobe analyses (to a 300 parts per million level as structure similar to that reported for BaCuO, <sup>8</sup> but with important determined by calibration scans). The results indicate a cubic number of microcrystals isolated from a partially melted pellet this material. The formula unit derived from the structure the Cu:Ba atomic ratio close to a value of 1.4. differences.

Superconducting transitions were established with onset near 90K in two isolated crystals about 50  $\mu m$  x 50  $\mu m$  100  $\mu m$  of the thalliumdoped barium cuprate phase that had been first examined in an X-ray diffractometer and found to be a pure cubic single crystal. The tech-

state due to inter-domain Josephson junctions. The temperature depen-T, onset temperature decreases from 60K to 70K for the 1:2:3 sample to the 1:2:4 sample. However, the differences are small and quantitative magnetometry measurements using both a SQUID and a force magnetometer expulsion and exclusion approaches 50 to 60% of ideal diamagnetism at magnetization versus field curve we estimate the lower critical field by electron microprobe analyses shows that the II content is lower in occurs (depressing  $T_c$  and decreasing the superconducting fraction) on onset of field-modulated microwave absorption in the superconducting phenomenon has now been widely investigated in the new high T oxide superconductors  $^{9}$  . Although a relatively strong microwave absorption signal relative to the mass of the sample was observed there may not superconducting phase present. We have also performed the microwave from the data in Fig. 5 it is also evident that superconductivity in 30K for the 1:2:4 sample. Comparison of the T1 content distribution state in this phase has spin glass-like character. In addition, the nique used to detect superconductivity in such small samples is the dence of the absorption for the crystals is shown in Fig. 4. This evidence of non-resonant microwave absorption at low temperatures. were performed on samples prepared from Tl:Ba:Cu = 1:2:3 and 1:2:4 5K indicating that bulk superconductivity occurs in these samples. numbers cannot be quoted at this time. We also found that TI loss fractions via powder X-ray diffraction. The results at different to be -100 G. The above results suggest that the superconducting magnetic fields for these samples are shown in Fig. 5. The flux the doped phase is very sensitive to the magnetic field. From a experiment on the undoped barium cuprate precursor and found no precursors, which showed majority thallium-doped barium cuprate be a direct relationship between absorption and the amount of aging of the samples in air.

AC and OC four-probe transport measurements were attempted on as-prepared pellets from the 1:2:3 and 1:2:4 precursors. Both samples showed strong metallic behavior down to our measurement limit of -10K. Zero resistance was not achieved but kinks in the data at 70 and 30K in the 1:2:3 and 1:2:4 precursor prepared sample respectively, were observed. It is possible that the percolative path was in these

samples is blocked by excess thallium much in the same way as excess potassium blocks the transport path in K-doped BaBio $_3^{\ 7}$ . Future work should determine the conditions of re-annealing the as-prepared pellets so that transport properties of the thallium-doped barium cuprate phase can be measured.

We believe at this time that thallium doping results in the closure of the semiconducting gap in BaCu0<sub>2</sub> and concomitant suppression of its known ferromagnetic interactions 10, to give a glass-like superconductor. Some remnant ferromagnetic character may, however, be still present as evident from the presence of reproducible oscillations near 40K in the susceptibility data (indicated by arrows in Fig. Sa).

We would like to thank Dr. J. Ferraro of the Molecular Structure Corporation for performing the crystal structure analyses and Dr. R. Antrim of Allied-Signal for obtaining the extensive microprobe data on these samples. The research at the University of California, San Diego, was partially supported by grants from NSF-CMR-13858 and ONR-NO0014-87-0338.

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### IABLE 1

Atomic coordinates, thermal factors (B) and occupancies for the cubic thallium doped barium cuprate phase at R  $^{+}$  0.05.

<u>Nаme</u> Вал Т11
0.0000
0.0000 0.2025 0.4317
0.2668
0.3438

# Figure Captions

- Fig. 1 Powder X-ray diffraction of pellets prepared from precursors: (a) 11:Ba:Cu  $\sim$  1:2:3 using  ${\rm Tl}_2{\rm Co}_3$ , and (b) 2:2:3 using  ${\rm Tl}_2{\rm Co}_3$ . Asterisks indicate peaks associated with the minority thallium rich phase.
- Fig. 2 Unit cell of the thallium doped barium cuprate showing the Cu(1), O(2) and (3) and thallium atoms along the body
- Fig. 3 (100) and (111) structure images and corresponding electron diffraction patterns (marked a and b respectively) of a crystal of the thallium-doped barium cuprate phase.
- fig. 4 Plots of the microwave absorption signal versus temperature for two single crystals of the thallium-doped barium cuprate phase. A background scan is also shown. The data was taken while warming the sample, which typically results in the sample temperature being approximately 5K cooler than the temperature indicated in the plot. Note that the temperature scales are different for each plot and non-linear.
- Fig. 5 (a) Zero-field cooled DC magnetization data taken in a force magnetometer at fields of 3 and 6 Oe for two separate preparations from a Ti:Ba:Cu = 1:2:3 precursor.

  (b) Zero-field cooled and field-cooled (Meissner) DC magnetization SQUID magnetometer data for a Ti:Ba:Cu = 1:2:4 precursor-prepared sample measured in fields of 1.8 and 8.1 Oe.

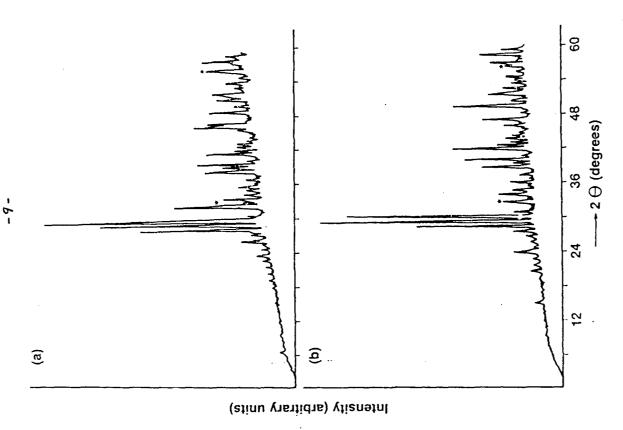
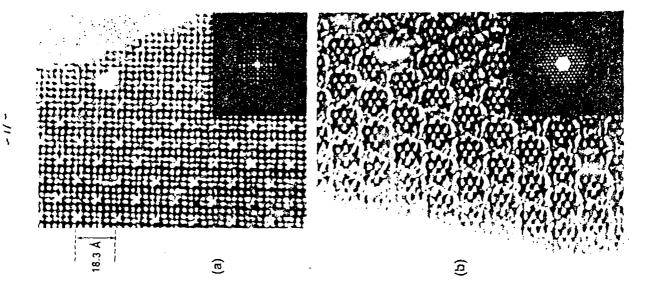
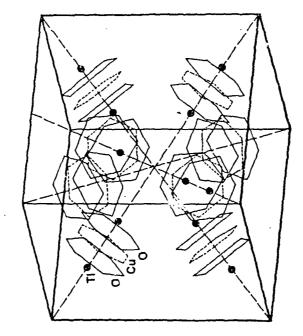


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